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(3) Hair stengthening and permanent waving composition.

© Composition for strengthening hair and for imparting a permanent configuration thereto comprising an alkyltrialkoxysilane and a solvent.

EP 0 159 628 A2

HAIR STRENGTHENING AND PERMANENT WAVING COMPOSITION

This invention relates to hair strengthening and permanent waving compositions, and more particularly, to reactive silicone hair strengthening and waving compositions.

Hair care is one of the most important parts of beauty care, and a large variety of hair treatment products are used for providing such care in the form of pre-shampoo conditioners, shampoos, after-shampoo conditioners, rinses, setting lotions, sprays, dyes, bleaches, permanent wave agents and the like. These products in addition to providing the desired result in the hair, such as luster, curl 10 combability, softness, color and an overall appealing look, also do damage to the hair, especially when used indescriminantly without professional guidance. Damage to hair may also result from other sources, such as combing, humidity, dryness, dirt, sunrays, such as u.v. and infrared 15 radiation, and pollution in the atmosphere. However, damage to hair occurs mostly in the form of physical and chemical changes in hair as a result of bleaching, oxidative dying, hair relaxing via alkaline relaxers and reducing waving and

20 curling preparations.

Hair damage may occur slowly and in various ways with frequent "treatment" of the hair. Over an extended time period the use of various chemicals in the hair treatment products will effect a weakened hair structure. of this undesirable effect is that which occurs when the natural configuration of hair is altered from curled to straight or vice versa. The hair is subjected to the action of a composition which relaxes the hair by rupturing the disulfide bonds in the keratin of the hair to produce free sulfhydryl groups. This breakage in the disulfide diminishes

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the rigidity of the hair proteins, leaving the hair pliable and soft. The compositions most commonly used as relaxers are based on sodium hydroxide, sulfite, or thioglycolate. When a thioglycolate based composition is used as the relaxer, the hair is treated with an oxidant, such as peroxide, to impart rigidity to the hair. These operations, particularly the reduction step, can be carried out at either room or elevated temperature. Both types of treatment, the cold as well as the hot, require visual evaluation of the hair. Ultimately, the quality of the hair, straightened or curled, rests on the subjective judgement of the salon operator, and more often than not because of the subjective element, the hair is either underprocessed or overprocessed.

or overprocessed, the hair subjected to oxidative hair preparations, lacks the desired texture, it is raspy, troublesome to comb, and require further conditioning. To correct these conditions some manufacturers of hair treatment products frequently supply post-treatment conditioners usually as part of a kit included with the acidic oxidative preparation. Others incorporate specific hair conditioning agents compatible with the oxidizing agents, such as non-ionic and cationic agents.

Hair conditioning agents are used not only in conjunction or after treatment with oxidizing agents but also to generally assist in the control and management of hair as for example after shampooing. Conditioned hair is easily untangled and combed through after shampooing, lays orderly when dry and provides a favorable feeling to the touch. The conditioning action on hair, particularly by cationic conditioning agents, is believed to be caused by the attraction of the positively charged agent to the negative sites on hair protein resulting in the deposition of the agent on to the hair fiber.

Cationic agents or surfactants have been used extensively as hair conditioning agents in creme rinses and shampoos. Best results have been obtained with cationic surfactants that are long chain high molecular weight quaternary compounds or long chain fatty amine salts. The positive charge of the quaternary surfactant is attracted to the negatively charged surface of the hair protein giving it lubricity during wet combing and a desirable texture after drying.

Other types of agents for deposition on hair used in the prior art are the organo silicones. The organo silicones, having hydrolyzable groups on the silicon atom, hydrolyze to the corresponding silanol and condense to an organo-silicone polymer on the hair fibers coating the same and providing body thereto are used for both conditioning and setting the hair.

While the above mentioned conditioning agents provide substantial benefits to hair, none is directed to strengthening the hair structure and thereby restore the hair, as much as possible, to its natural strength.

The foregoing discussion illustrates some of the problems associated with hair care and solutions provided therefor by the prior art.

The present invention addresses the problem of treating the weakened hair, whether such condition is caused by nature, the environment or hair treatment preparations applied thereto.

The present invention relates to a composition for strengthening the tensile strength of hair comprising

a, an alkyltrialkoxysilane having the formula

$$R_1$$
-Si $\underset{OR_2}{\underbrace{OR_2}}$

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wherein

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 R_1 is an aliphatic radical of C_1 - C_{18} carbon atoms, such as methyl, ethyl, propyl, butyl, hexyl, octyl, decyl, octadecyl; substituted aliphatic radicals selected from the group consisting of 3-aminopropyl,

N,N-diethyl-3-aminopropyl, and N-(propyl) urea; and an alkenyl radical, such as vinyl and allyl;

 $\rm R_2$ is an aliphatic radical of $\rm C_1$ - $\rm C_4$ carbon atoms, such as methyl, ethyl, propyl and butyl;

b, a solvent for said alkyltrialkoxysilane. The solvent may consist of water, ethanol, isopropyl alcohol, nonoxynol-4, or an aqueous solution of ethanol, isopropyl alcohol or nonoxynol-4.

invention, a solution is prepared by adding an alkyltrialkoxysilane to the appropriate solvent with mixing. The composition preferably comprises about 0.5 to 40% v/v and more preferably 2 to 10% v/v of an alkyltrialkoxysilane in the solvent. The preferred solvent is an aqueous solution of ethanol, isopropyl alcohol or nonoxynol-4 in which the water constitutes no more than about 20% v/v of the total solvent volume.

Although the silanes described herein are stable for several days in alcohol/water phases having from 0 - 20% water, it is preferred to prepare the solution just prior to application to the hair. If the solution is allowed to stand in the presence of water for prolonged time periods, excessive polymerization and premature precipitation of the silane may occur, thus rendering the composition less effective for its intended use.

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The method of application of the composition includes applying the composition to the hair by submerging the clean hair in the composition or applying the composition with an applicator and working it into the hair with the fingers and a comb. The process is completed by blow drying the hair with hot air.

The present invention is based on the discovery that the reactive alkyltrialkoxysilane containing three labile alkoxy groups in the presence of water hydrolyzes to form three reactive silanol groups which in turn condense 10 with each other to form oligomers. The oligomers then hydrogen bond with functional groups containing active hydrogens (hydroxyl, amino, carboxyl, sulfhydryl and the like) in the hair. During drying or curing a stable covalent bond is formed with the hair with concomitant loss of water. 15 While the curing process will occur at room temperature, it is preferred to speed up the reaction by drying the hair with a hot air dryer. The application of heat speeds up evaporation of the solvent and the by-products of the reaction. Once water has been removed from the hair, the 20 resulting bonds become stable to hydrolytic action. degree and rate of polymerization of the silane is determined by the amount of water available and the organic substituent of the silane.

This characteristic of the silanes of the present invention allows for their application to hair for increasing hair strength, retaining hair configuration such as curls, and providing manageability to hair.

The silanes of the present invention are either commercially available or can be prepared by methods known in the art using commercially available starting materials and reagents.

Silanes are readily solubilized in ethanol, 1 isopropyl alcohol and many other organic solvents, however, for the present invention the preferred solvent for the silanes is an aqueous/alcohol mixture containing up to 20% by volume of water. This composition ensures that a sufficient 5 amount of water is available to hydrolyze the silane and possibly swell the hair fibers for increased penetrations of the active ingredient prior to the polymerization step. Silanes show unexpected stability to water in such vehicles as determined by the time to hazing (precipitation of-10 siloxane polymers). For example, solutions of ethyltriethoxysilane dissolved in 80/20 ethanol/water remain clear for several days. As the percentage by volume of water increases beyond 20% of the total solvent composition, more rapid hazing and precipitation of the siloxane polymer is 15 observed to occur, thus rendering the composition less effective for its intended use.

Test methods used in connection with the invention and working examples follow.

Tensile Strength

This test relates to the determination of the force required to break (tensile strength at breakpoint) the hair sample by using a constant elongation rate tester such as the Instron Tensile Tester. The test is performed as follows:

Each tress of hair to be tested is conditioned in a 50% relative humidity chamber for at least 24 hrs prior to use. Twenty strands of hair, randomly selected from each tress, are used for the test. A two-inch long section is cut from each hair strand and weighed to the nearest 0.1 microgram. The weighed cut sections are placed into distilled water and allowed to hydrate for at least 16 hours.

- Each hydrated hair sample is placed between the jaws of the Instron Tensile Tester, which are preset at 1 inch apart, and clamped securely. The tensile strength is then determined according to the manufacturer's instructions.
- The tensile strength is the force required to break the hair strand. This force is expressed in terms of grams force per cross sectional area of hair strand.

Method For Determination of Silane Content in Hair

The silicon content of treated hair is determined colorimetrically as silicomolybdate or as molybdenum blue and 10 the result calculated as silane. Approximately 0.7 gram ± 0.1 mg of hair is weighed accurately into a beaker, to which 5 ml of concentrated nitric acid is added. The sample is heated to dryness and the addition of nitric acid and heating steps are repeated until a white residue remains in the 15 beaker. Then 10 ml of 1:10 hydrochloric acid is added to the beaker and the content is filtered with suction through a membrane filter. The beaker is thoroughly washed with 1:10 hydrochloric acid and the liquid is filtered. The filter is placed in the bottom of a polyethylene beaker and 1.0 ml of 20 hydrofluoric acid is added to cover the filter. The beaker is then covered and allowed to stand for 30 min. of water and 50 ml of boric acid solution is added with stirring, and the solution is heated at 40°C in a water bath for at least 10 min. Four (4) ml of molybdic acid reagent is 25 added while stirring. Then 20 min. later, 20 ml of 10N sulfuric acid is added with stirring. If any yellow color persists, the solution is read within 2 min. after acidification in a spectrophotometer at 420 nm against distilled water. If a colorless solution results, the 30 solution is allowed to stand for 2 to 5 min. and 1 ml of 1-amino-2-napththol-4-sulfonic acid reagent is added, which is then mixed and read after 20 min. at 820 nm.

a calibration curve in the yellow silicomolybdate concentration range (420 nm) is made by diluting 0.5, 1, 2 and 3 ml aliquots of the stock silicon (lmg/ml) standard to 25 ml in polyethylene beakers and proceeding as hereinbefore described from and including the step of heating the solution for at least 10 min. After completing the steps as indicated, absorbance is read at 420 nm. Absorbance against mg silicon is then plotted. A calibration curve is also prepared in the molybdenum blue concentration range (820 nm) by diluting 50, 100, 200 and 500 microliters of the stlicon stock standard (lmg/ml) to 25 ml in polyethylene beakers and proceeding as described above for the silicomolybdate calibration curve. Absorbance is read at 820 nm.

The concentration of silicon is obtained from the appropriate calibration curve. The calculation is as follows:

Percent silane = $\frac{\text{mg silicon}}{\text{grams of hair}} \times \frac{\text{mole weight of silane}}{\text{atomic weight of silicon}} \times 0.1$

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EXAMPLE 1

- A hair sample previously weakened by sodium hydroxide was submerged in 3.0% v/v solution of ethyltriethoxysilane (obtained from Petrarch Systems, Inc., Levittown, PA) in a mixture of 80/20 ethanol/water. The hair was rinsed free of excess silane and cured by blow drying with a hot air dryer for about ten minutes. The hair was tested using the above-described test procedures. The results were as follows:
- a. The hair treated with sodium hydroxide solution showed 11 to 14% decrease in tensile strength;
 - b. The sodium hydroxide weakened hair which subsequently was treated with the silane-containing composition increased in tensile strength back to within 97 to 98% of its original (untreated) tensile strength;
 - c. The silane treated sodium hydroxide weakened hair picked up between 0.4 to 1.4% silicon by weight, calculated as ethyltriethoxysilane.

EXAMPLE 2

20 nonoxynol-4 was prepared. Immediately prior to treating the hair, 40 parts of the silane/nonoxynol-4 phase was added with stirring to 60 parts of water. The hair, which was previously weakened by sodium hydroxide was completely wetted with the silane/nonoxynol-4/water solution. The solution was allowed to remain on the hair for 30 minutes. The hair was rinsed free of excess solution and blow dried with a hot air drier.

Upon testing, the data showed a 10% increase in tensile strength of the hair.

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EXAMPLE 3

1 A swatch of virgin hair was heavily damaged by a 45 minute treatment with a 2% aqueous solution of sodium The alkali-damaged swatch was washed with a pH 7.0 shampoo to restore the acid balance, allowed to dry under 5 ambient conditions and subdivided into a series of equal-sized tresses. Three tresses were selected and each treated for 30 minutes with 10 grams of the silane-containing composition of Example 2, cured and blow dried. procedure used to treat the three tresses was exactly_the 10 same except for the manner in which the excess silane was Instron analyses were carried out on all of the The result and the manner or removing excess silane are shown in Table I.

Table I

Instron Results for Prepared Tresses

	Sample	<u>n</u>	Tensile X*	Strength (G) SEM**
20	Control virgin hair	20	29.49	<u>+</u> 0.61
	Control alkali-damaged hair	20	14.48	<u>+</u> 1.07
	Alkali-damaged hair, silane treated, rinsed, then cured	20	17.51	<u>+</u> 0.56
25	Alkali-damaged hair, silane treated, cured, then rinsed	20	16.56	<u>+</u> 0.59
30	Alkali-damaged hair silane treated, cured, then rinsed and shampooed	: 20	16.95	<u>+</u> 0.702

n denotes the number of determinations here and in subsequent tables.

 $[\]underline{X}^*$ denotes the mean of the number of measurements here and in subsequent tables.

SEM** denotes the standard error of the mean here and in subsequent tables.

The data in Table I show that all the silane treated tresses have significantly increased in strength (about 15-20%) relative to the alkali-relaxed control. No significant difference in hair strength is shown among the silane-treated tresses indicating that the mode by which excess reagent is removed from the hair has no affect on hair strength.

It was also discovered that if hair, arranged in a desired configuration with curling rollers or rods, is treated with a silane-containing composition of the present invention and blow dried, the silane bonding to the hair not only increases hair strength but also maintains the hair in the shape in which it was dried. This effect is shown for both alkali-weakened and virgin hair. The resulting permanent wave resists the tendency to revert to the initial configuration even after repeated shampooing.

EXAMPLE 4

A dark brown virgin hair swatch obtained from a single lot was washed with a 10% sodium lauryl sulfate solution, rinsed with tap water, and blow dried at room The swatch was subdivided into eighteen tresses temperature. ten inches long each weighing one gram. One tress was selected to serve as virgin control and five other were set aside for silane treatment. The remaining twelve tresses were set aside for the alkali treatment. Before treatment, each tress was gently combed and separately rolled on curling Alkali treatment was carried out by submerging the rolled tresses in a 2.0% sodium hydroxide solution for one Subsequently, each tress was rinsed under running tap water for at least 10 minutes. The rolled tresses were towel dried, soaked in pH 7.0 buffer for one hour to restore the acid balance, and rinsed out thoroughly to remove the buffer. The rolled tresses were finally dried at room temperature.

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Equimolar solutions of silanes denoted in Tables II 1 and III were prepared separately in a mixture of 80:20 ethanol/H2O, to carry out silylation. The virgin and alkali treated tresses on rollers were treated separately for 30 minutes with 100 ml of 0.26M solution of silane. The rolled tresses were blow dried for four minutes after the silane treatment with a 1600-watt blow drier and thoroughly sprayed with water. The rolled tresses were allowed to stand for five minutes and finally blow dried for 15 minutes at the high temperature setting. Unsilylated virgin and alkali 10 control were also subjected to the same treatment. The hair tresses, after equilibrating to room temperature, were carefully unwound from the curling rods and mounted on the curl retention board. The mounted specimens were then incubated at 100°F/95% relative humidity. Curl retention 15 data were obtained periodically over 24 hours. The tresses were removed from the board, wetted under running tap water for 1 minute, dried and soaked in a beaker of water for 30 additional minutes, removed, air dried, twice shampooed and rinsed again. The final curl length and patterns of each 20 tress were evaluated to determine curl stability. comments are summarized in Tables II and III.

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1			pattern	ern	ern	wave	
5			and S-wave pattern	S-wave pattern e	good S-wave pattern shine	curl and S-wave	
10	<u>ir</u>	Comments	Good curl No shine	Very good S Good shine	Very good Good shine	Very good curl pattern	
15	Table II Pre-Treated Hair	SEM	+1.35	<u>+</u> 0.13	+0.20	+0.42	(0.26м)
20	Tab Alkali Pre-	% Curl Retention (X)	12.27	14.55	17.80	16.36	f silanes (0.26M)
	ΨI	cl	5	2	2	2	equal moles of
25		ne (1)					
		Silane Conc. (%)	0.00	10.84	4.70	06.9	(l) All solutions contain
30			ali	ane	1 ane	urea	utions
			Control (alkali relaxed)	Octadecyl triethoxysilane	3-Aminopropyl triethoxysilane	N-(triethoxy silylpropyl)urea	All sol
35		Sample	Control relaxed)	Octadecyl triethoxy	3-Ami triet	N-(cr silyl	(1)

(1) All solutions contain equal moles of silanes (0.26M)

5	·	Comments	Almost no curl, no S-wave pattern	Very good body wave	Fair body wave	Good body wave	Good body wave	Fair body wave	_
		3	Al. Pa	Ve	Ŗ	3	99	r. B	
15	- III Hair	% Curl Retention (x)	7.16	12.73	11.04	11.35	11.94	10.38	
	Table - III Virgin Hair	·	•						
20		c۱	-	-	7	-	7	-	
25		Silane (1) Conc. (%) .	00.0	10.84	7.20	4.70	. 06.4	06.90	-
30		Sample	Control virgin hair	Octadecyltriethoxy- silane	Octyltriethoxy- silane	3-Aminopropyltri- ethoxysilane	Vinyltriethoxy- silane	N-(triethoxy- silylpropyl)urea	
0.5									

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EXAMPLE 5

1 Series of tresses were prepared from a single lot of dark brown virgin hair, each tress being 1.5 ± 0.1g in weight and 10" long. Two of the tresses, selected at random to serve as permanent waved controls, were treated with 5 Collagen Amino Acid Professional Formula (active ingredients: 9.2% ammonium thioglycolate, 10% sodium bromate). Fourteen virgin hair tresses were wrapped on rods and immersed for 1 hour in a 2% sodium hydroxide solution. The wrapped tresses, after having been rinsed free of alkali, 10 were treated with pH 7.0 buffer to restore the acid balance The tresses were rinsed free of buffer and to the hair. treated as described in Tables IV and V. The procedure used for the evaluation and curing of alkali-treated hair was described in Example 4. Heat was also applied to the control 15 samples for an identical period of time. All the prepared tresses were allowed to equilibrate under ambient conditions of temperature and humidity.

20 additional minutes, removed and shampooed twice the Flex Shampoo Roy Tinsed free of soap and allowed to air dry. The final curl length and pattern of each tress were carefully evaluated to determine curl stability.

Results are shown in Tables IV and V.

						-	16-			.: 01 .: .:
1			no S-wave	ern	ern	er E	ב ב			
5		nts	Little curl - no	ра	Good S-wave pattern	Good S-wave pattern	Good S-wave pattern	Very good S-wave pattern	Very tight curl	Very good S-wave pattern
10		Comments	Littl	od S poog	S poog	S poop	S poog	Very goo pattern	Very t	Very go pattern
15	u	SEM	ı	i	ı	t	1	±0.20	+0.20	09.0+
	Table - IV Curl Retention	% Curl Retention (X)	10.2	15.4	15.0	13.4	14.2	15.2	14.1	16.7
20	Cur	ci	-	1	1	-		2	. 2	2
25				7	$_{ m S1}$	elled-30%	elled-30%	sl in	ıl in	α.
30			Alkali relaxed control	relaxed/5% ETES1	relaxed/30% ETES1	Alkali relaxed/urea swelled-30% ETES1	Alkali relaxed/salt swelled-30% ETESl	Alkali relaxed/30% ETES ¹ DET ²	Alkali relaxed/30% ETES1 DMF3	Permanent waved controls
35		Sample	Alkali	Alkali	Alkali	Alkali ETES	Alkali ETES ^l	Alkali r DEF2	Alkali r DMF3	Permaner

(1) ETES = Ethyltriethoxysilane in 80/20 alcohol/water in here and in Table V

⁽²⁾ DET = Diethyltoluamide

⁽³⁾ DMF = Dimethylformamide

ı '	Comments	Very little curl, no S-wave pattern	Good body wave	Good body wave	Good body wave	Very little curl, no S-wave pattern	Good S-wave pattern	Good S-wave pattern	Fair S-wave pattern
	ं ।	Ver S-w	Coo	Goo	000	Ver S-w	Goo	009	Fai
10	SEM	09.0+	+0.80	0+T	0+1	0+1.	0+1	0+1	+1.00
Table - V	% Curl Retention (x)	.6*9	12.2	12.6	12.6	7.5	12.2	11.4	10.4
20	⊆ l	2	. 7	2	2	2	2	2	2
25		ol	Sa	swelled	swelled	ntrol	ETES	ea swelled	lt swelled
30]e	Virgin hair control	Virgin hair/5% ETES	Virgin hair/salt swelled 5% ETES ¹	Virgin hair/urea swelled	Alkali relaxed control	Alkali relaxed/5% ETES	Alkali relaxed/urea swelled 5% ETES	Alkali relaxed/salt swelled 5% ETES
35	Sample	Vire	Virg	Vire 5% E	V; rę 5% r	Alka	Alka	Alks 5% E	Alke 5% E

Hair was swelled with a 2.8M LiCL/0.5M Na $_250_4$ aqueous solution prior to treating the hair with silane. Ξ

Hair was swelled with a 4M aqueous solution of urea prior to treating the hair with silane. (2)

Table IV demonstrates that silylation of alkali treated hair on curling rods produces a curl pattern equivalent to that produced by a conventional permanent waving treatment, and is substantially greater than the curl produced in the alkaline relaxed control. Curl "memory" was retained in the alkali-silylated tresses even after repeated shampooing.

Table V demonstrates that silane compositions of the present invention impart a good stable curl to alkali treated hair and, in addition, that the curl retention of the virgin hair is significantly improved after silylation through the formation of a stable body wave pattern.

It will be understood that modifications and variations may be made without departing from the spirit and scope of the novel concept of the present invention.

WHAT IS CLAIMED IS:

- 1. A composition for strengthening the tensile strength of hair comprising:
 - a., an alkyltrialkoxysilane having the formula

$$R_1 - Si \qquad \underbrace{OR_2}_{OR_2}$$

wherein

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- R₁ is an aliphatic radical of C₁ C₁₈ carbon atoms; substituted aliphatic radicals selected from the group consisting of 3-aminopropyl, N,N-diethyl-3-aminopropyl, N-(propyl)urea and alkenyl;
- $$\rm R_2$$ is an aliphatic radical of $\rm C_1$ $\rm C_4$ carbon atoms; and
 - b., a solvent for said alkyltrialkoxysilane.
 - 2. The composition of claim 1 wherein the aliphatic radical is methyl, ethyl, propyl, butyl, hexyl, octyl, decyl or octadecyl.
 - 3. The composition of claim 1 or 2 wherein the alkenyl is vinyl or allyl.
 - 4. The composition of any of claims 1 to 3 wherein the solvent is water, ethanol, isopropyl alcohol or nonoxynol-4.
- 5. The composition of any of claims 1 to 3 wherein the solvent is an aqueous solution containing ethanol, isopropyl alcohol or nonoxynol-4.
 - 6. The composition of clam 5 wherein water constitutes no more than about 20% v/v of the solvent.
- 7. The composition of any of claims 1 to 6 wherein the alkyltrialkoxysilane is present in about 0.5 to 40% v/v based on the total volume of the composition.

- 8. The composition of any of claims 1 to 7 wherein based on the total volume of the composition alkyltrialkoxysilane is present in an amount from about 2 to 10% v/v in 98-90% v/v of a solvent.
- 9. The composition of any of claims 1 to 8 wherein the alkyltrialkoxysilane is octadecyltriethoxysilane, n-octyltriethoxysilane, 3-aminopropyltriethoxysilane, vinyltriethoxysilane, n-(triethoxysilylpropyl) urea or ethltriethoxysilane.
- 10. A method of strengthening hair comprising the steps of:
 - a., applying to the hair an effective amount of the composition of any of claims 1 to 9
 - b., applying heat to the hair to effect curing.
- 11. The method of claim 10 wherein the hair is an alkali-weakened hair.
 - 12. The method of claim 10 or 11 wherein the hair is virgin hair.
- 13. The method of any of claims 10 to 12 wherein prior to step (a) the hair is set to the desired configuration.
 - 14. The method of any of claims 10 to 13 wherein the hair is pre-treated with an alkali or a thioglycolate prior to applying the composition.

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